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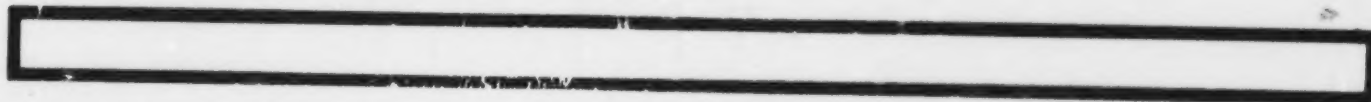
Hydrocarbon Solvent Recovery in the
Presence of Resin Contaminants

Arkansas Univ., Fayetteville

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HYDROCARBON SOLVENT RECOVERY IN THE PRESENCE
OF RESIN CONTAMINANTS

by

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FOREWORD

When energy and material resources are extracted, processed, converted, and used the related pollutional impacts on our environment and even on our health often require that new increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report describes a recently completed project. The purpose of the project was to develop a process which will economically recover and recycle a hydrocarbon solvent from a gaseous stream in which there are suspended particles such as resins. The system selected for study was recovery of acetone from an air stream in which these are suspended epoxy resin particles. The developed process is applicable to other such gas solvent-particle systems encountered in industry.

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ABSTRACT

A system was developed to recover acetone from an air stream in which there were suspended epoxy resin particles. This recovery problem is encountered in the manufacture of fiber glass reinforced plastic pipe. It is representative of numerous other industrial situations which require the recovery of hydrocarbon solvents from a gaseous stream containing resin particles in order to eliminate atmospheric pollution. For example, the printed circuit board industry utilizes over 20 million pounds annually of the identical epoxy-ketone resin system utilized by pipe manufacturers. In addition, over 30 million pounds annually of these epoxy resins are utilized in the tool, casting and molding industries.

The system developed was a three-stage low temperature condensation process preceded by a cascade impactor. A scale model of the system was designed and constructed. It was tested in the laboratory, and on a split stream of an actual plant process.

Roughly 95 percent of the resin particles were removed in the impactor. The first stage condenser operated at 42°F, and removed the residual resin particles and roughly 80 percent of the water brought in with the ambient air. The second stage operated at -31°F and achieved residual water removal. Acetone of 99 percent purity was recovered in the third stage operating at -85°F.

A full scale system was designed to process 3,800 cfm of air containing 0.92 (vol) percent acetone and 1.25×10^{-5} lb/ft³ of resin particles. The fixed capital investment for this system was estimated to be \$758,000.

The developed impactor-condenser system is a technically feasible process. Use of the impactor in conjunction with one-, two-, or three-stage condensation may be considered as a possible alternative in any solvent recovery application. An economic evaluation will be required for each potential application, with the final decision to utilize the process being based on the economics of the specific recovery problem.

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SECTION 1 INTRODUCTION

Hydrocarbon solvents are utilized in many industries in processing schemes which require recovery of these solvents from a gaseous stream in order to eliminate atmospheric pollution. Much is known about solvent recovery, and it is practiced widely in the chemical industries. However, recovery solutions are complicated in instances where certain contaminants are present in the discharge stream.

One process for manufacturing reinforced plastic pipe utilizes acetone as a solvent for the resin. The gaseous discharge from the process is an acetone-contaminated air stream, also containing resin particles and certain other chemical decomposition products. These resin particles plug conventional recovery equipment such as adsorption beds, filters, etc. Numerous other examples of this type of recovery problem are found in the plastics industry, especially in the production and utilization of epoxy resins.

The magnitude and scope of the problem in the use of epoxy resins is ever-expanding. Approximately 315 million pounds of epoxy resins were produced in the United States in 1979 (1,2). Many of the epoxy resin manufacturing operations utilize a hydrocarbon ketone as a solvent for the resin. Comprising the majority of these ketone-epoxy resin applications are three major industrial manufacturing classifications--epoxy pipe, electrical laminates (printed circuit board), and pre-impregnated articles.

There are nine major plastic pipe fabricators in the United States, with at least five of these companies marketing epoxy resin piping. Approximately 19 million pounds of epoxy resin were utilized by the pipe industry in 1978, while the printed circuit board industry utilized approximately 20 million pounds of the identical epoxy resin which was utilized by pipe manufacturers (3). The third major industrial classification utilizing epoxy resins is the pre-impregnation process, including tool, casting, and molding industries. Also in 1978, 31 million pounds of epoxy resins were utilized by these broad-based industries (3).

Less than ten years ago the liquid epoxy utilized by the protective coating and bonding and adhesive industries comprised 80-85% of the total epoxy market. Today, however, the solid and semi-solid epoxy system utilized by pipe, printed circuit board, and pre-impregnation industries account for 25% (70 million pound/year) of the epoxy market. Projections reveal an ever increasing market for all epoxies with an accelerated portion being allotted for solid and semi-solid systems.

It is concluded from these examples and figures that the problem of recovery of hydrocarbons in the presence of resin contaminants is potentially very large. Extensive consultation with leading manufacturers of recovery and clean-up equipment reveals that commercially available off-the-shelf systems which are satisfactory are not available. Thus, the objectives of this research project were:

- (1) to develop a system which will economically recover a solvent from a gaseous stream in which there are suspended contaminants such as resins,
- (2) to construct and test a scale model of the recovery system, and
- (3) to design, specify equipment, and estimate installed costs for a full-scale recovery system.

The A. O. Smith-Inland (AOS-I) Inc. manufactures fiber glass reinforced plastic pipe at their Little Rock, Arkansas, plant. In the process, glass fibers are pulled through a vat of epoxy resin and acetone solvent where they are impregnated with the resin. The resin-coated fibers travel from the vat over hot drums.

A continuous strip of fiber glass reinforced plastic (which is later processed into plastic pipe) is formed on the drums. The solvent acetone is vaporized from the hot drums and it is picked up by a blower-induced air stream passing up over the drums, into the hood covering the tape-machine, and is exhausted from the process. The violent vaporization of the acetone on the hot drum surface erupts resin particles into the air stream where they are captured and ultimately transported into the exhaust ductwork. Ambient water vapor enters with the air stream.

AOS-I had previously installed an acetone recovery system which included a water scrubber, parallel carbon adsorption beds, and a distillation column. This unit became permanently inoperative after only a very short time. Due to the low efficiency of the water scrubber, resin carry-over plugged the adsorption beds and corroded the distillation unit. Scrubber screens and spray nozzles were also plugged with epoxy.

Because this acetone-resin system is representative of other such systems, and because of the demonstrated difficulty in dealing with this system, it was chosen for study in this project. Also, the results of this study are applicable to others in the industry. The acetone concentration in the air stream is well below the lower explosive limit, and it is expected that other such processes would exhibit air stream concentrations comparable to these values. It is likely that these air streams are currently being discharged directly to the atmosphere by most, if not all, of the industry.

Because of their vested interest in the project, A. O. Smith provided significant project funds in a cooperative funding agreement with EPA. The Company also supported the project by providing facilities for plant testing of the scale-model recovery system developed during the project.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

A cascade impactor combined with a three-stage low temperature condensation system is a technically feasible processing system for recovery of acetone from an air stream containing epoxy resin particles. The flat plate cascade impactor removed more than 95 percent of the resin particles. The remaining resin particles and the bulk of the ambient water vapor were removed in stage one, operating at 42°F. Stage two, operating at -31°F, achieved residual water removal. Acetone of 99 percent purity was removed in the third stage operating at -85°F.

The full-scale system designed for this particular application to process 3,800 cfm of air containing 0.92 (vol) percent acetone had a fixed capital investment of \$758,000. Components of this investment figure are shown in Table 15.

It is recommended that the condensation process utilizing one, two, or three stages as needed, be considered as a possible alternative in any solvent recovery application, with the final decision on utilization of the process based on the economics of the specific recovery problem.

SECTION 3 PRELIMINARY CONSIDERATIONS

The problem addressed in this research project is the recovery of a hydrocarbon solvent in the presence of resin contaminants. A specific instance of this general problem area selected for study was the recovery of acetone from an acetone-contaminated air stream which also contains epoxy resin particles. This particular stream is a major discharge stream from the fiber-glass-reinforced plastic pipe manufacturing operation at the A. O. Smith-Inland Inc. plant at Little Rock, Arkansas.

Prior to initiation of this research project, there was significant effort by AOS-I to recover the acetone (4). The results of their preliminary work are summarized in this Section.

Several alternatives were considered. Incineration of the gaseous stream is a potential solution. However, it is expensive and energy inefficient since for solvent concentrations in the air stream below the spontaneous combustible level, auxiliary fuel would be required. For concentrations above this level, an explosion and fire hazard result. Extensive burner controls would be required to maintain a combustible mixture. Finally there are significant risks that non-volatile resin particles in stack emissions could be extensive thus requiring costly collection equipment in the stack in order to meet EPA standards.

Water spray scrubbers were rejected because of their low collection efficiency (50-85%), their high probability of becoming plugged with the resin, their need for a follow-up waste water treatment system, and their addition of undesired moisture into the air stream. Scrubbers utilizing liquids such as hot caustic would also have a low collection efficiency and would be susceptible to plugging.

Bag, panel, and column filters were rejected because of their high probability of becoming plugged with the resin after only short periods of operation. The Linde Molecular Sieve Division advised that molecular sieves would not be acceptable, also because of rapid plugging.

After considering these and several other alternatives, it was concluded (4) that the optimum system for acetone recovery would be the use of a cascade impactor to remove the bulk of the resin particle contaminants, followed by a liquid bath/packed bed for solvent removal from the gaseous stream. Solids could be settled, skimmed, or filtered from the liquid, and the solvent could be recovered by a separation process, probably distillation in most applications.

Published data on impactors is scant. However, one set of data provided by the Brinks Company indicated that a low-cost, simple design, flat plate cascade impactor could remove up to 98 percent of the resin particles. The particles in this test ranged from 0.08 to 200 microns and were representative of the actual process stream. This compares to 50 percent removal from the actual stream in a water scrubber test.

The remaining resin particles would be removed in a liquid bath/packed bed which would also condense the solvent from the gaseous stream. This bed was envisioned as consisting of an inert packing material covered by a pool of liquid. The liquid would be circulated through a refrigeration system to maintain a low temperature for condensation of the acetone. The condensed acetone would be filtered for final removal of resin particles, and separated for recycle by distillation. This was the status as the current cooperative research project was initiated.

SECTION 4 EXPERIMENTAL PROGRAM

The research project was initiated based on the conclusions of the preliminary study as outlined in the previous section. The experimental program was divided into two parts, the impactor phase concerned with removal of resin contaminants and the condenser phase concerned with recovery of the acetone.

Equipment was constructed to produce a process stream representative of the actual plant process stream at AOS-I. Based on obtaining laboratory equipment of a reasonable size, an air flow of 90 cfm was established as a design basis for the model. This results in a model to plant scale-up ratio of 1:42 for the anticipated 3,800 cfm plant flow.

The scale model impactor and condenser and associated equipment were also designed and constructed. This was followed by experimental testing, utilizing the simulated process stream. The model equipment was then re-located to the Little Rock plant. Extensive experimental testing was done at the plant site, utilizing a split-stream of the main process stream diverted to the recovery system.

a. Impactor Phase

Included in this phase of the project is not only the impactor but also the equipment for generation of the simulated process stream. Air flow to the system was provided by a Schwitzer Model 325 blower which delivered 125 cfm of air at a 4 psig discharge pressure. Other major items of equipment which were designed and built were the particle generation chamber, the impactor, and the particle filter.

The particle generation chamber was a 9-inch by 9-inch sheet metal duct fitted with flanges at either end and on top for attachment of the system piping. Air flow from the blower was directed through the chamber. An entry port was provided on top of the chamber and a plexiglas window was installed on the side for visual access.

Initial plans were to establish the desired acetone and resin concentrations in the air stream by dripping an acetone-resin mixture onto an electric hot plate located inside the chamber. It was anticipated that the boiling action of the acetone on the hot plate would transport resin particles into the air stream as occurs on the hot drum surface in the actual process.

The hot plate was constructed using two 500-watt heating strips. A copper plate with lips along each side was attached to the top of the heating strips. A thermocouple was inserted between the strips and the copper plate. The hot plate was bolted to the duct at only two points so that its angle of inclination could be varied. An angle of 5° with horizontal appeared to provide an adequate compromise between retention time and resin run-off.

In the actual plant process, the hot forming drum temperature is 450°F, and the air velocity past the boiling surface is near 200 feet per minute. For these conditions, one to three grams of resin particles are transported into the air stream per pound of resin fed to the process.

These conditions of temperature and velocity were duplicated in the particle generation chamber. Resin mixtures consistent with the actual process were used. Although violent boiling occurred on the surface of the hot plate, resin particles were not detected downstream. Several different arrangements of baffles were used in order to increase the air stream velocity past the boiling surface. Velocities as high as 1,440 ft per minute and temperatures as high as 700°F were utilized in an effort to generate particles, but without success.

In order to more closely duplicate the actual process, fiber glass filaments were stretched across the hot plate, and the resin mix was dripped onto this fiber glass material. Again, good boiling was achieved with violent eruptions of acetone from the plate. However, a maximum of 0.05 grams of particles per pound of resin were transported into the air stream compared to the 1-3 grams per pound in the actual process. Since this concentration was too low to produce meaningful results in the impactor tests to follow, the generation of resin particles by the hot plate method was abandoned.

A decision was made to attempt particle generation by an atomizing spray nozzle. The atomizer nozzle was located inside the original chamber, above the hot plate, so that the particles generated could be suspended in the air stream. The hot plate was left in the chamber for later use in vaporization of acetone for testing of the condenser.

Provisions were made to control the flow rate and pressure of both the resin and the atomizing air supply. By manipulation of these four variables, it was possible to produce a wide range of particle sizes, from a "fog" of micron size particles to "liquid droplets" of several hundred microns in size. Initial atomizer tests indicated that up to 50 grams of particles per pound of resin could be suspended in the air stream. This amount was more than satisfactory for testing of the impactor.

The preliminary work by AOS-I indicated that 90-98 percent of the resin contaminants could be removed from the air stream by a low-cost, flat plate, cascade impactor. Since no mechanical design information was available, it was necessary to include as much flexibility as possible in the model impactor design. Provision was made for variation of the number of plates, the spacing between plates, the plate-to-bottom clearance, and the slot dimen-

sions of the plate, the latter by fabricating plates of various dimensions. Provision was not made for varying the slope of the impactor, since it was felt that this was not critical as long as the slope was sufficient to provide satisfactory drainage of the collected resin.

A filter was also designed for installation downstream from the impactor so that impactor collection efficiency could be ascertained. An orifice meter was built and located downstream from the filter for measurement of system flow rates.

The test procedure for evaluation of the impactor was as follows. With the impactor removed from the system and the filter located adjacent to the particle generation chamber, a flow of 90 cfm of air was established through the system. Flow of resin mix to the atomizer nozzle was initiated. The desired spray pattern was obtained by adjusting pressures of the resin and air stream to the nozzle.

After only a few minutes of operation, the air flow would begin to decrease as a result of progressive filter plugging. Continued operation would result ultimately in a completely plugged filter with a zero air flow. It was observed after each test that the upstream duct from the filter was coated with resin deposits while the downstream duct was clean.

The impactor was then installed in the same location as the filter had been in the previous tests, and the filter was attached to the downstream side of the impactor. Using the same air flow rate and atomizer variable settings as before, impactor tests were made.

From flow rate data, direct weighing of the filter cloth, and material balances, the collection efficiency of the impactor was established. After each test, the impactor system was opened and cleaned. Resin was not observed in the short section of duct between the impactor and the filter. Inspection of the impactor revealed resin build-up on the first four blades and much lesser amounts on the fifth and sixth blades from the impactor inlet. Drainage from the impactor was good. Quantitative results are presented in the next section.

b. Condenser Phase

At the time of initiation of this research project, the concept of the condenser was a single stage liquid bath (i.e. a flooded packed bed) operating at a temperature of 41°F. Acetone and ambient water vapor would be condensed from the air stream in the liquid pool along with collection of the remaining resin particles which escape the impactor. Recovery of the acetone from the water would be accomplished by distillation.

However, initial vapor-liquid calculations revealed that a liquid pool temperature of 41°F was not feasible. Because of its relatively high vapor pressure at this temperature, acetone would be vaporized from the pool rather

than condensed. In order to achieve adequate acetone removal from the air stream, a pool temperature of -85°F to -100°F would be required.

Thus, the single stage condenser concept was abandoned. At this point, there was a significant and unforeseen delay in the condenser phase of the project as a search for a viable processing scheme was initiated. Several alternatives were considered with preliminary calculations being required on each.

In a parallel search, A. O. Smith Research and Development conceived a three-stage condenser for recovery of the acetone. The first stage would operate with a water pool at $42-50^{\circ}\text{F}$ to remove roughly 80 percent of the water plus the residual resin particles. The second stage would operate with a glycol-water mixture at -31°F to remove essentially all of the remaining water. The final stage would operate with a liquid acetone pool at -85°F to remove up to 95 percent of the acetone at a greater than 98 percent purity, thereby eliminating the need for a distillation step for acetone recovery.

Preliminary quotes had been obtained early in the project during the single stage calculations for refrigeration equipment to remove the necessary heat at -85°F . A 3.5 ton unit required for the scale model test work was quoted at \$60,000 while a purchased price of \$385,000 was quoted for a 70-ton unit to accommodate the total plant flow. Although this latter figure was only preliminary, it provided an indication that while low temperature recovery of acetone was technically feasible, in-depth economic evaluation of the process would be required.

Subsequent conversations of A. O. Smith Research and Development with another supplier of low temperature refrigeration indicated that the required equipment would cost substantially less than the initial quote. A decision was made to proceed with the three-stage concept and to build a scale model unit to verify the concept.

However, the refrigeration equipment required for the scale model unit was prohibitively expensive. To circumvent this problem, a surplus room air conditioner was modified for first-stage cooling, and plans were made to achieve the very low stage two and stage three temperatures by the use of liquid nitrogen.

Design calculations were completed and the three-stage unit was constructed. A 55-gallon barrel with a removable top was utilized for each of the three stages. The process air stream from the impactor was sparged into the bottom of stage one and then was passed overhead and sparged into the bottom of the next two stages, in order.

A grid was installed near the bottom of stage one, above the sparger, for support of a couple of inches of raschig ring packing. The cooling coil was removed from a room air conditioner and the coil was relocated inside stage one, above the packing. Flexible refrigerant hoses were used to connect the coil to permit movement into and from the stage.

Each of the stages two and three were equipped with a finned coil for the liquid nitrogen coolant. The coils were piped so that liquid nitrogen passed through the stage three coil and the stage two coil in series, and then was sparged directly into the stage two pool, exiting with the process air stream from stage two. Provisions were also made to introduce liquid nitrogen to the stage two coil as a mixture with the gaseous nitrogen discharge from the stage three coil. In this manner, the stage two and stage three liquid pool temperatures could be controlled independently.

Thermocouples were located in the liquid pool, in the exit air stream, in the coil inlet, and in the coil outlet for each of the three stages. The air temperature at the impactor exit was measured with a thermometer. Sample ports were located in the vapor space and in the liquid pool of each stage.

From stage three, the process air stream passed through a section of straight pipe and an orifice meter before exiting the process. By shutting off two of the three flows in order, the process air flow, the nitrogen flow to stage two, and the nitrogen flow to stage three, each could be measured separately. A thermocouple was located at the orifice meter for measurement of the metering temperature.

Styrofoam insulation of 1½-inch, 3-inch, and 4-inch thickness was applied to stages one, two, and three, respectively. Connecting piping was also insulated. Each stage was equipped with a drain line. A framework was built for support of the three stage condenser equipment. The stages were assembled for testing in the laboratory with testing of the total system to follow at the plant site.

Because of the high cost of liquid nitrogen cooling, stages two and three were not put on stream until preliminary test work had been done on stage one. Stage one was filled with water to a depth of 22 inches (11 inches of freeboard), and a flow of 90 cfm of air was established. There was substantial entrainment of liquid water droplets. A demister screen was installed. With a freeboard of 18 inches, an air flow of 68 cfm was achieved with no carry-over. Demister screens were then installed in stages two and three.

Several tests were made at air flows of 55-60 cfm. For entering temperatures near 100°F, the stage one exit temperatures were from 36°F to 42°F. However, icing of the coil occurred as the 36°F exit temperature was realized. More satisfactory operation was achieved with a pool temperature of 35°F. No icing occurred and the exit temperature was 42°F. This appeared to be a good combination to use for design. This affords a 3° margin of safety above freezing and a 7° difference for heat transfer, which is adequate.

Stage one was also tested with resin input to the system. Resin loadings of 4-5 times actual plant loadings were used. The approximately 5 percent resin which escaped the impactor was collected as a less-dense-than-water scum in the first stage. It was not sticky, and it could be removed from the system with few complications. There was some slight accumulation of the scum on the barrel walls and on the outer edges of the demister screen.

These deposits were also easily removed. There was no carry-over of resin from the first stage.

Stages two and three were then put on-stream with liquid nitrogen cooling for preliminary testing. Stage two was loaded with a 60-40 percent (wt) mixture of ethylene glycol and water to a depth of 14 inches (19 inches freeboard). Stage three was loaded to a depth of 14 inches with acetone. The desired temperatures of -31°F and -85°F were achieved in stages two and three, respectively, at process air flows up to 50 cfm.

At this point the fluid flow and heat transfer problems of the system had been resolved. It has been established that the system would operate at process air flows up to 50 cfm, with 19 inches of freeboard in each stage, with no carry-over of liquid from one stage to the next. It had also been established that there would be complete entrapment in stage one of residual resin particles escaping the impactor. And, finally, it had been established that the desired pool and exit air temperatures could be achieved in each stage.

Remaining to be established were the mass transfer characteristics of the system. This was to be conducted at the laboratory site using an available gas chromatograph for analysis of the vapor streams and the liquid pools. A limited number of these tests were made indicating adequate water removal in stage one and very good three-stage acetone removal.

However, because the project had fallen behind schedule due to the reasons given above, the scale-model recovery system was moved to the plant site prematurely. Establishment of the mass transfer characteristics proved more difficult, expensive, and time-consuming because the lack of a gas chromatograph at the plant site required the contracting and scheduling of an outside laboratory firm.

The total system, including the impactor, was assembled at the plant site and a split-stream of the actual process stream was diverted to the test equipment. After the elimination of mechanical problems, the system was operated for extended periods once or twice weekly for two months. Quantitative results are presented in the next section.

SECTION 5

RESULTS AND DISCUSSION

The experimental program described in the previous section produced quantitative results which were used to evaluate the impactor and the three-stage condenser system.

A summary of the impactor test data is included as Table 1. Each entry in the table represents several trials, and the data were reproducible. Resin removal efficiencies of ca. 95 percent were common with the maximum being about 97 percent for the six-blade case at a 1/2-inch blade spacing. Impactor pressure drops were approximately three inches of water with a maximum of 3.4 inches, also occurring for the six-blade case at a 1/2-inch blade spacing. Drainage of the resin from the impactor was good. Some build-up of resin does occur on the blades, but the impactor will operate for extended periods without plugging.

From the test data, a linear velocity of 2,000 ft/min in the baffle slots appeared to be a feasible compromise value for the design basis for the full-scale impactor. Lower velocities reduce the collection efficiency while pressure drop increases as the velocity is increased.

Results from selected condenser trials are included as Tables 2-11. For many of these trials, the total system was not operated, and complete system data are not reported. This was done to reduce operating costs, since many of the trials were for testing only specific functions or specific parts of the experimental apparatus. The complete system was operated for extended periods on a limited basis, and these data are reported in Tables 10 and 11.

Condenser trial 2, reported in Table 2, was made to demonstrate the condensation capability of the three-stage system. The concentration of both acetone and water was reduced from three percent (wt) to only a trace in the exit air stream. Stage three was operated at -115°F in this test. After 18 hours of cumulative operation, the acetone concentration in the stage one liquid pool had increased to 2.8 percent (wt). Table 3 also illustrates an increase in acetone concentration from 0.52 percent to 0.63 percent during a four-hour period of operation. It also shows an increase in water concentration in the stage three pool from one percent to 1.7 percent. Because the extremely cold temperatures produced frost in the sample port, it is felt that these samples may have become contaminated.

Stable operation with pool temperatures near the desired values was achieved in condenser trial 6, reported in Table 4. Again, there was a condensation of acetone in the stages one and two liquid pools. Acetone concentration in the stage three exit vapor was below the 2,200 ppm (wt basis)

TABLE 1
SUMMARY OF IMPACTOR TEST DATA

<u>No. of Baffles</u>	<u>Baffle Spacing, in.</u>	<u>Impactor Pressure Drop inches of water</u>	<u>Resin Removal Efficiency, %</u>
2	1/2	2.2	93.7
	3/4	2.2	95.1
	1	1.8	94.3
4	1/2	2.7	95.8
	3/4	2.7	96.2
	1	2.5	94.2
6	1/2	3.4	97.2
	3/4	3.1	96.2
	1	2.9	96.5

TABLE 2
CONDENSER TRIAL 2 RESULTS - 1/19/82

<u>Stage</u>		Elapsed Time	
		<u>2 Hrs</u>	<u>18 Hrs</u>
inlet	Acetone conc, wt%	3.2	
	Water conc, wt %	3.0	
1	Exit air temp, °F	36	
	Pool acetone conc, wt%	0	2.8
3	Exit air temp, °F	-115	
	Exit air acetone conc, ppm	trace	
	Exit air water conc, ppm	trace	

TABLE 3
CONDENSER TRIAL 5 RESULTS - 2/24/82

<u>Stage</u>		Elapsed Time	
		<u>0 Hrs</u>	<u>4 Hrs</u>
inlet	Acetone conc, wt%	0.5*	0.5*
1	Pool temp, °F	46	
	Pool acetone conc, wt%	0.52	0.63
2	Exit air temp, °F		-32
	Pool water conc, wt%	43	45
	Pool glycol conc, wt%	57	55
3	Pool temp, °F		-101
	Exit air temp, °F		-100
	Pool water conc, wt%	1.0	1.7

*Estimated from corresponding operation on another date

TABLE 4
CONDENSER TRIAL 6 RESULTS - 3/4/82

<u>Stage</u>		<u>Elapsed Time</u>		
		<u>0 Hrs</u>	<u>1 Hrs</u>	<u>4 Hrs</u>
inlet	Acetone conc, wt%	0.5	0.5	
1	Pool temp, °F		46	
	Pool acetone conc, wt %	0.0		0.76
	Exit air acetone conc, wt %		0.18	
2	Pool temp, °F		-21	
	Exit air temp, °F		-18	
	Pool acetone conc, wt%	0.087		0.78
	Pool water conc, wt%	36		40
	Pool glycol conc, wt%	64		60
	Exit air acetone conc, wt%		0.023	
3	Pool temp, °F		-86	
	Exit air temp, °F		-82	
	Pool water conc, wt%	0.6		2.1
	Exit air acetone conc, ppm (wt)		1600	

design concentration at exit temperature near the -85°F design temperature. This 2,200 ppm (wt) exit vapor concentration was a condition of the emission permit granted by the State Department of Pollution Control and Ecology.

Data to this point indicated that the acetone could be recovered in the condenser system and that the exit air stream would meet the design specifications. However, the concern became that the acetone would be condensed in stages one and two rather than in stage three.

Several condenser trials were made to alleviate this concern. Tables 5 and 6 include the results of condenser trials 7 and 8, respectively. An acetone concentration of 18.8 percent (wt) was charged to the stage one water pool, and stage one was operated for an extended period. At the end of eight hours, the concentration had decreased to 0.9 percent. Operation was continued and, after an additional eight hours, the acetone concentration had decreased to 0.028 percent.

This same behavior was exhibited by the glycol-water pool as shown by the results in Tables 7, 8, and 9. Acetone concentrations of roughly eleven percent were established in the stage two liquid pool. For reasons unknown, the concentration had decreased to only ten percent after five hours of operation in condenser trial 12. However, in trials 10 and 11, after only three hours of operation, the concentration had decreased to eight percent and four percent, respectively. These results indicate that a dynamic steady state will be reached at some low acetone concentration in each liquid pool which will be acceptable for plant operation.

Tests of the complete impactor-condenser system were made in trials 13 and 14 reported in Tables 10 and 11, respectively. The desired temperatures were maintained in each stage, and an extended period of steady-state operation was achieved. It is noted that the stage two exit air temperature was lower than the pool temperature in these tests. This was due to the liquid nitrogen being sparged to the stage two pool. There was good resin removal in the impactor, and no resin carry-over from the stage one condenser.

The stage one results verified the previous test results of less than one percent acetone in the pool at dynamic steady-state. Stage two results indicate that a steady-state concentration of 3-4 percent acetone would be experienced in the glycol-water pool. It should be noted that these concentrations are for current plant operation with roughly 0.5 percent acetone in the process air stream. For the proposed plant operation, the acetone concentration will increase by a factor of near two. Although not a linear relationship, it is expected that the acetone concentrations in the stages one and two pools will increase by a corresponding amount.

Analysis of the exit air stream from stage three verified the acetone removal capability of the condenser system. A value of 3,800 ppm (wt) acetone in trial 13 was measured before the desired stage three temperature was reached. A value of 2,400 ppm (wt) acetone was determined in trial 14.

TABLE 5
CONDENSER TRIAL 7 RESULTS - 3/10/82

<u>Stage</u>		Elapsed Time			
		<u>0</u>	<u>1.75 Hr</u>	<u>2.4 Hr</u>	<u>7.6 Hr</u>
1	Pool acetone conc, wt%	18.8	12.0	8.75	0.9

TABLE 6
CONDENSER TRIAL 8 RESULTS - 3/11/82

<u>Stage</u>		<u>0 Hr</u>	Elapsed Time	
			<u>3.25 Hr</u>	<u>7.9 Hr</u>
1	Pool temp, °F		46	54
	Pool acetone conc, wt%	0.79	0.051	0.028

TABLE 7
CONDENSER TRIAL 10 RESULTS - 3/15/82

<u>Stage</u>		Elapsed Time		
		<u>1 Hr</u>	<u>2 Hr</u>	<u>3 Hr</u>
1	Pool temp, °F	46	46	48
	Pool acetone conc, wt%	0.19	0.17	0.12
2	Pool temp, °F	-28	-10	-19
	Exit air temp, °F	-23	-4	-16
	Pool acetone conc, wt%	11.4	10.4	8.2

TABLE 8
CONDENSER TRIAL 11 RESULTS - 3/16/82

<u>Stage</u>		Elapsed Time		
		<u>1 Hr</u>	<u>2 Hr</u>	<u>3 Hr</u>
1	Pool temp, °F	46	52	51
	Exit air temp, °F	52	55	59
2	Pool temp, °F	-19	-33	-34
	Exit air temp, °F	-17	-28	-32
	Pool acetone conc, wt%	11.0	3.8	4.3
3	Pool temp, °F		-78	-90
	Exit air temp, °F		-73	-88

TABLE 9
CONDENSER TRIAL 12 RESULTS - 3/24/82

<u>Stage</u>		Elapsed Time		
		<u>1 Hr</u>	<u>3 Hr</u>	<u>5 Hr</u>
1	Pool temp, °F	47	38	41
	Exit air temp, °F	52	47	51
	Pool acetone conc, wt%	1.5	1.1	0.8
2	Pool temp, °F	-21	-31	-36
	Exit air temp, °F	-22	-32	-38
	Pool acetone conc, wt%	11.8	11.5	9.9
3	Pool temp, °F	-84	-88	-95
	Exit air temp, °F	-78	-84	-92

TABLE 10
CONDENSER TRIAL 13 RESULTS - 4/2/82

<u>Stage</u>		<u>Elapsed Time</u>			
		<u>0 Hr</u>	<u>1 Hr</u>	<u>2 Hr</u>	<u>4 Hr</u>
1	Pool temp, °F		43	41	42
	Exit air temp, °F		53	47	53
	Pool acetone conc, wt%	0.7	0.6		0.3
	Exit air acetone conc, wt%			0.44	
2	Pool temp, °F		-35	-37	-30
	Exit air temp, °F		-36	-39	-34
	Pool acetone conc, wt%	4.0	3.8	3.9	3.3
	Pool glycol conc, wt%	59.0	59.0	60.3	57.1
	Exit air acetone conc, wt%			0.27	
3	Pool temp, °F			-63	-91
	Exit air temp, °F			-58	-87
	Exit air acetone conc, ppm (wt)			3,800	

TABLE 11
CONDENSER TRIAL 14 RESULTS - 4/14/82

Stage		Elapsed Time			
		<u>0 Hr</u>	<u>1 Hr</u>	<u>3 Hr</u>	<u>5 Hr</u>
1	Pool temp, °F			42	48
	Exit air temp, °F			48	53
	Pool acetone conc, wt%	0.2		0.8	
	Exit air acetone conc, wt%				0.96
2	Pool temp, °F		-27	-32	-30
	Exit Air temp, °F		-30	-32	-32
	Pool acetone conc, wt%	3.3	2.5	2.5	2.9
	Pool glycol conc, wt%		57		56.4
	Exit air acetone conc, wt%				0.42
3	Pool temp, °F		-89	-94	-95
	Exit Air temp, °F		-83	-89	-89
	Exit air acetone conc, ppm (wt)				2,400

Final quantitative information provided by the experimental program was heat transfer coefficients for each of the three stages. These were calculated from experimental flow rate-temperature measurements taken during steady-state operation of the equipment. Both coil-to-pool surface coefficients and pool-to-air-stream volumetric coefficients were determined. These are presented in Table 12.

The conclusion drawn from the experimental program is that low temperature condensation is a technically viable process for recovery of the acetone. The required heat transfer, mass transfer, and component separation can be achieved in three stages. Specific processing schemes and equipment requirements are discussed in the next section.

During the period of in-plant testing of the experimental unit, acetone concentration of the process split-stream was 5,000 ppm (wt). A reduction to 1,600 ppm (wt) in the effluent translates to a 68 percent recovery of the acetone. However, proposed plant operation anticipates 20,000 ppm (wt) acetone in the plant effluent. Reduction to 1,600 ppm (wt) will result in a 90 percent recovery of the acetone to the condensation unit. Acetone purity of greater than 98.5 percent is expected from stage three.

TABLE 12
EXPERIMENTAL HEAT TRANSFER COEFFICIENTS

	Surface Coil-to-pool <u>Btu/hr ft² °F</u>	Volumetric pool-to-air <u>Btu/hr ft³ °F</u>
Stage 1	2.0	21.2
	2.3	17.0
Avg.	$\frac{3.2}{2.5}$	$\frac{19.8}{19.3}$
Stage 2	-	30.2
	8.7	29.2
Avg.	$\frac{1.4}{-}$	$\frac{24.5}{28.0}$
Stage 3	-	30.9
	2.5	39.0
Avg.	$\frac{2.9}{2.7}$	$\frac{29.7}{33.2}$

SECTION 6 PROCESS DESIGN

a. Impactor Design

The results of scale-model impactor tests were used to design the impactor. The critical design parameter is a 2,000 ft per minute linear velocity through the slots of the impactor. Design sketches for the impactor are included as Figures A-1 to A-6 of the Appendix.

The impactor was divided into three shells arranged in parallel. For design capacity, all three impactors would be on-stream. For lower flows, one or two impactors would be on-stream as needed. Also, the impactors were designed to allow two impactors to accommodate the total plant flow in emergency situations.

A simple flat-blade design was used. Blades were designed to be easily and cost effectively fabricated. Initial installation and subsequent replacement of blades should also be simple.

b. Three-stage condenser Design

The experimental program demonstrated the validity of the three-stage low temperature condensation process for recovery of the acetone. This system results in high purity acetone being recovered for recycle without distillation. A considerable savings is thus realized by elimination of the energy-intensive distillation operation.

Several different types of equipment can be utilized to achieve the three-stage recovery. The required heat and mass transfer can occur in liquid baths as were utilized in this experimental program, in countercurrent flow packed beds, or in finned-tube heat exchangers. Calculations were made for each in order to compare the three processes.

Preliminary design bases were chosen, material and energy balances were completed, and the equipment items were sized. It is noted that these preliminary bases were used for the initial equipment comparison, and that subsequent changes were made which are included later in this report. A summary of the initial equipment comparison is shown in Table 13.

Initial calculations revealed this problem to be heat transfer rather than mass transfer limiting. That is, if equipment is provided which will obtain the desired temperatures in each stage exit, then the desired condensation will occur in each stage. The experimental volumetric heat transfer

TABLE 13
INITIAL EQUIPMENT COMPARISON

	Stage 1	Stage 2	Stage 3
DESIGN BASIS: 3000 CFM AIR FLOW @ 128°F			
Entering Air Humidity = 0.043 lbs/lb			
Entering Acetone Concentration = 1.7% (wt)			
Heat duty, Btu/hr	605,900	300,000	210,200
Exit temperature, °F	42	-31	-85
I. LIQUID BATH CONDENSER			
Pool Volume Required, ft ³	1,650	230	198
Vessel Dia. for 14" Liquid Depth, Ft.	42	15.8	14.7
Pressure Drop, each Stage (Approx.), Inches H ₂ O	14	14.7	11
II. PACKED COLUMN			
Liquid Circulated through External Heat Exchanger			
Column Diameter, Ft.	5.0	5.6	5.0
Column Height, Ft.	6.1	1.0	2.0
Pressure Drop, Inches H ₂ O/ft of Packing	0.4	0.4	0.4
III. FINNED TUBE HEAT EXCHANGER			
Coolant Circulated Inside 1" Tubes, 5/8" Fin Height X 10 fins/inch			
2 1/4" Triangular Pitch			
Tubes Required	240		96
Tube Length, Ft.	3		3
Rows of Tubes	12		6
Tubes Per Row	20		16
Bundle Width, Ft.	4		3
Fin Side Pressure drop, Inches H ₂ O	0.4		0.2

coefficients reported in Table 12 were used to size the liquid bath condensers. The resulting pool volumes were not excessively large. However, because a reasonable pressure drop necessitated a low liquid pool depth, a very large vessel diameter was required. This consideration eliminated the liquid bath as an alternative.

Countercurrent flow packed columns were sized for each of the three stages. These would be operated with the liquid in each stage circulated through an external heat exchanger cooled by the refrigerant. Column diameters of 5-6 ft were calculated with packed heights of 6 ft, 1 ft, and 2 ft being required for stages one, two, and three, respectively. Pressure drop was calculated to be only 0.4 inches of water per ft of packing in each of the three stages.

Another alternative would be to utilize finned tube exchangers cooled directly by the refrigerant. Results are reported in Table 13 for only stages one and three, with stage two being excluded because of the icing problem at -31°F. A stage two finned tube exchanger was included in later calculations with a parallel exchanger being provided so that cyclic defrosting of the exchangers could be done.

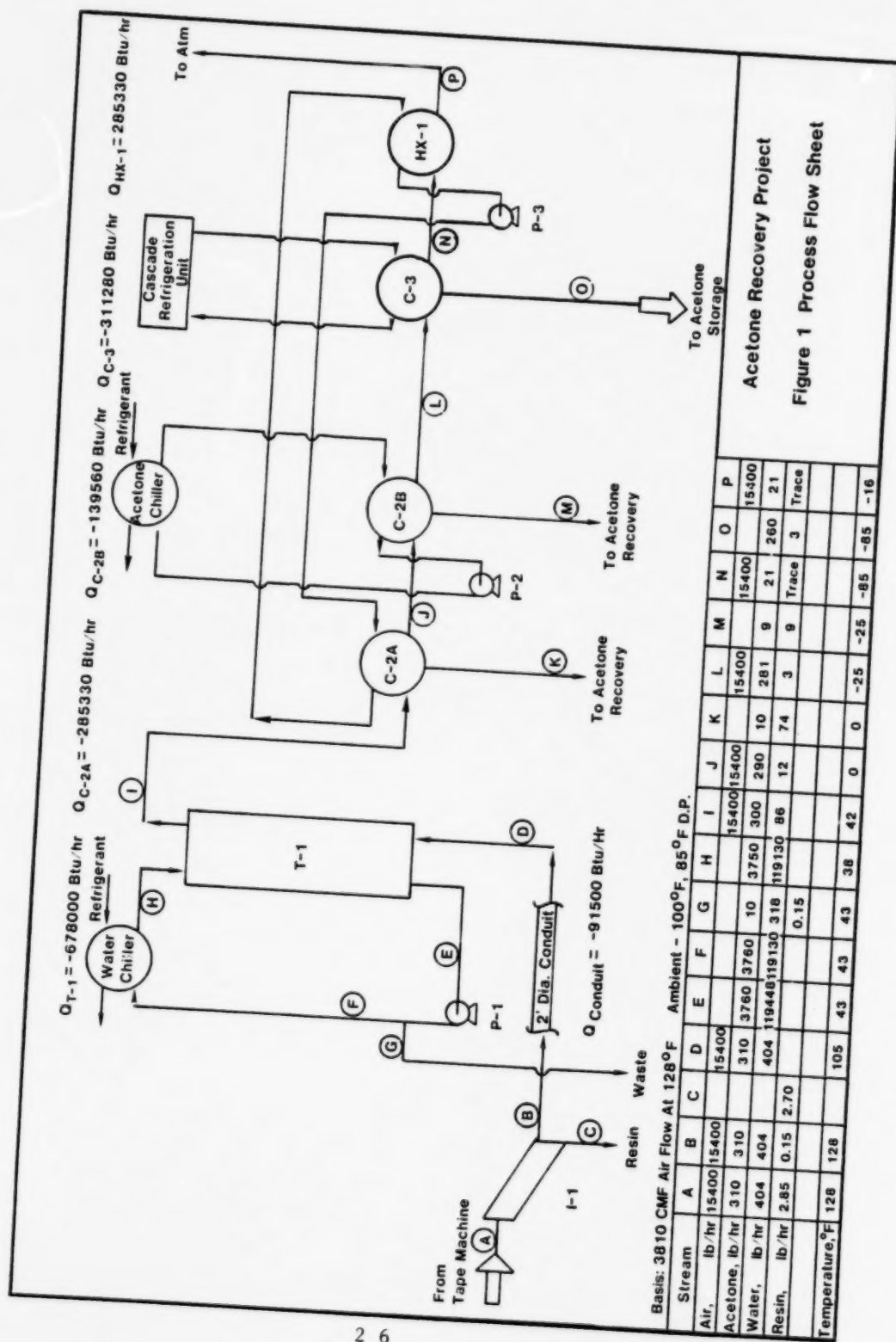
The conclusions drawn from these preliminary calculations are that either packed columns or finned exchangers would be satisfactory for stages two and three, with the decision to be based on the economics of the two systems. Stage one would require a packed bed with a few inches of liquid pool in the bottom for removal of the residual resin particles. A finned exchanger would not be acceptable for stage one because of its susceptibility to plugging by the resin.

Several changes were made in the design bases following these first-pass calculations. Also, several changes were evaluated in the processing sequence in an effort to get better heat integration. The finalized process flow sheet including a material and energy balance summary is included as Figure 1. An equipment list based on the flow sheet is included as Table 14.

Referring to Figure 1, flow from the tape machine passes through the impactor where 95 percent of the resin is removed and drained away. The remaining 5 percent is shown leaving with the tower T-1 waste stream. In actual operation, this residual resin would collect in the tower and be periodically flushed from the tower during process downtime.

The first stage packed tower was re-sized based on the finalized balances. A 6-ft diameter tower with a 4.5-ft packed section would be required. Water from the bottom of the tower is circulated through an external water chiller where its temperature is reduced from 43°F to 38°F and then is returned to the top of the tower. A purge stream removes the water plus 10 lb/hr of acetone condensed in tower T-1. Further processing of this purge stream is required because of its acetone content. Development of treatment methods for this stream was beyond the scope of this study.

A packed bed was used for stage two in initial calculations. To prevent icing, a mixture of water and ethylene glycol was circulated in the tower.



Acetone Recovery Project

Figure 1 Process Flow Sheet

TABLE 14
EQUIPMENT LIST

<u>Equipment No.</u>	<u>Service</u>	<u>Specifications</u>	<u>No. Req'd</u>
T-1	Water Removal Tower	Packed tower Diameter - 6 ft Height - 8.5 ft Packed Section - 4.5 ft Packing-1" Raschig Rings	1
C-2A	Stage Two Initial Condenser	Air-Acteone Exchanger Duty = 285,330 Btu/hr Lmtd = 30°F U = 5 Btu/hr ft ² °F A = 1,900 ft ²	2
C-2B	Stage Two Final Condenser	Air-Acteone Exchanger Duty = 139,560 Btu/hr Lmtd = 17°F U = 5 Btu/hr ft ² °F A = 1,650 ft ²	2
C-3	Stage Three Condenser	Air-Refrigerant Exchanger Duty = 311,280 Btu/hr Lmtd = 29°F U = 5 Btu/hr ft ² °F A = 2,150 ft ²	1
HX-1	Stage Three After Cooler	Air-Acetone Exchanger Duty = 285,330 Btu/hr Lmtd = 26°F U = 5 Btu/hr ft ² °F A = 2,200 ft ²	1
P-1	Stage One Circulation Pump	Water Pump Capacity - 240 cpm Head - 40 ft Motor - 5 Hp	2
P-2	Stage Two Circulation Pump	Acetone Pump Capacity - 97 cpm Head - 40 ft Motor - 2 Hp	2
P-2	Stage Two-Three Circulation Pump	Acetone Pump Capacity - 240 cpm Head - 40 ft Motor - 5 Hp	2

Water chiller, Acetone chiller, cascade refrigeration unit provided as part of packaged refrigeration unit.

This resulted in problems in glycol make-up and glycol recovery. It was thus decided to replace the packed bed with finned tube exchangers. A parallel exchanger would be necessary so that cyclic defrosting of the exchangers could be done.

In order to achieve some cooling from the final effluent, the stage two exchanger was divided into two shells in series. Cooling of the first, condenser C-2A, was by a circulating coolant being heat interchanged downstream with the final effluent. Cooling of the second stage two exchanger, condenser C-2B, was by external refrigeration. It is noted again that a parallel exchanger would be required for each of the condensers C-2A and C-2B for cyclic defrosting.

Several cases were calculated and it was determined that the optimum discharge temperatures were 0°F and -25°F, respectively, for the two condensers. This -25°F stage two discharge temperature differs slightly from the -31°F target value used in the experimental work.

Stage three condensation would also be achieved in a finned exchanger, condenser C-3, cooled by external refrigeration, rather than in a packed bed. The discharge temperature from this stage would remain at -85°F to achieve adequate acetone clean-up of the discharge stream. Acetone of near 99 percent purity is produced for recycle to the process.

From stage three, the cold air stream would be interchanged in heat exchanger HX-1 with the coolant circulating through the first of the stage two condensers. Atmospheric discharge from this exchanger would be at -16°F with acetone concentration of less than 1,400 ppm.

The process flow sheet and process description were submitted to several companies for a price quotation on the equipment. From these quotes, the total installed cost of the project was estimated to be \$758,000. Components of this investment figure are shown in Table 15.

In conclusion, the three-stage low temperature condensation process may be considered as a possible alternative in any solvent recovery application. Stage temperatures and other operating parameters are fixed by the particular system involved, by the specifications on the final effluent, and by the specifications on the recovered component. The process would be adapted to meet these specifications and the technical feasibility affirmed. It is probable that technical feasibility can be attained for most recovery problems by proper selection of operating parameters.

Material and energy balances would then be completed, equipment selected and sized, and the economics determined. The final decision for any potential application would be made at that point.

TABLE 15
FIXED CAPITAL INVESTMENT FOR IMPACTOR-CONDENSATION SYSTEM

<u>Item</u>		
1	Refrigeration equipment, heat exchangers, pumps, surge tanks, and controls	\$ 386,000
2	Packed column for Stage 1	8,000
3	Installation of Items 1 and 2	95,000
4	Impactors, high pressure blower, hood modifications and duct work	28,000
5	Holding tank and pumps	40,000
6	Distillation unit	25,000
7	Unit substation, starters, and primary wiring	60,000
8	Millwrights and field wiring	15,000
9	Insulated roofing	11,000
10	Drainage system	6,000
11	Lab testing equipment	15,000
		<u>\$ 689,000</u>
	10% Contingency	69,000
		<u>\$ 758,000</u>

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2. News brief, The Society of the Plastics Industry, Inc., Vol 10, Issue VI, August, 1979.
3. Modern Plastics, pg. 59, January, 1979.
4. Maynard, J. T., "Acetone Recovery," A. O. Smith Corporation, Research and Development Division, Internal Company Report AT-2, Jan. 25, 1979.

APPENDIX A
IMPACTOR DESIGN SKETCHES

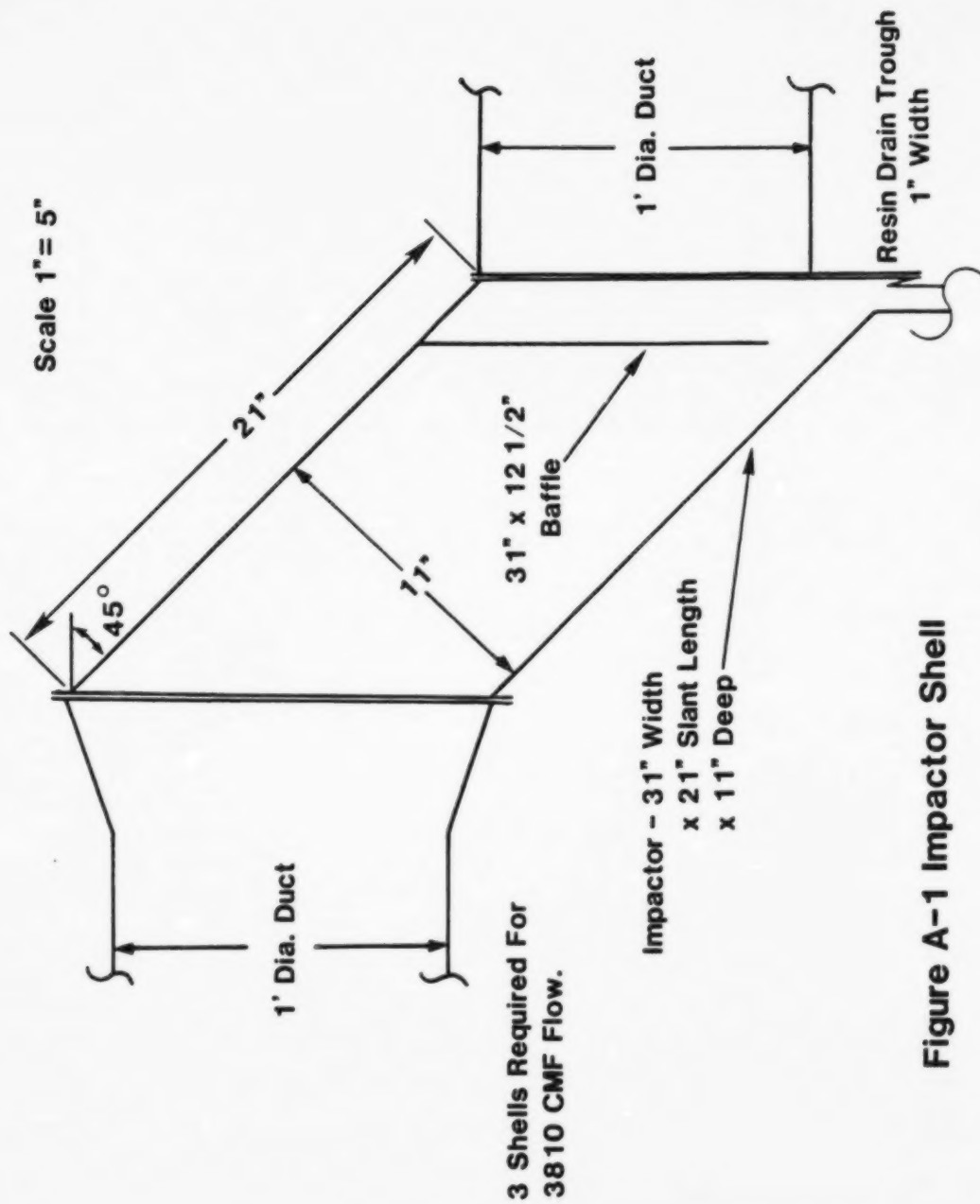


Figure A-1 Impactor Shell

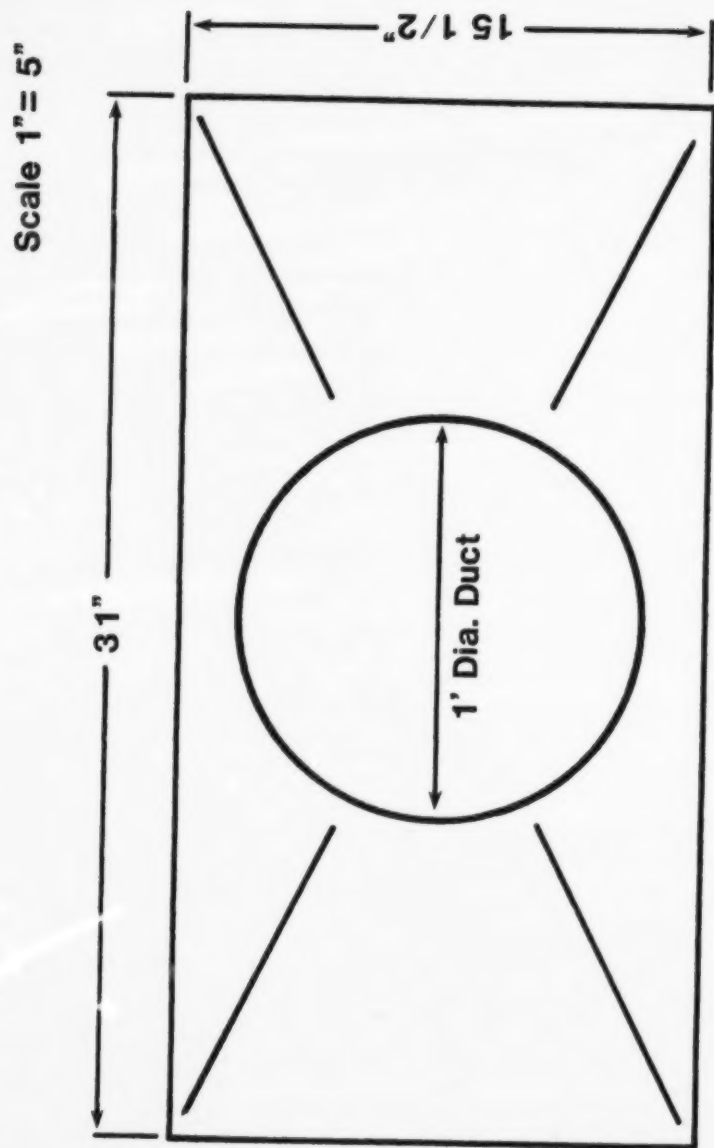


Figure A-2 Impactor Entrance

Scale 1" = 5"

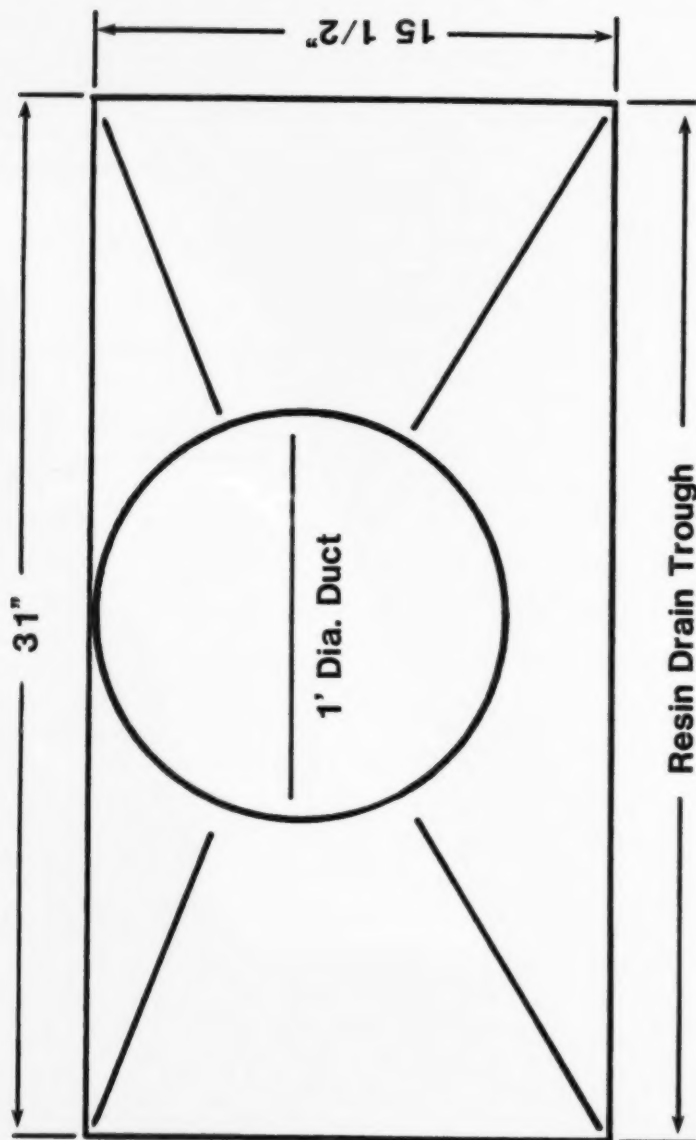


Figure A-3 Impactor Exit

Scale- 1" = 2 1/2"

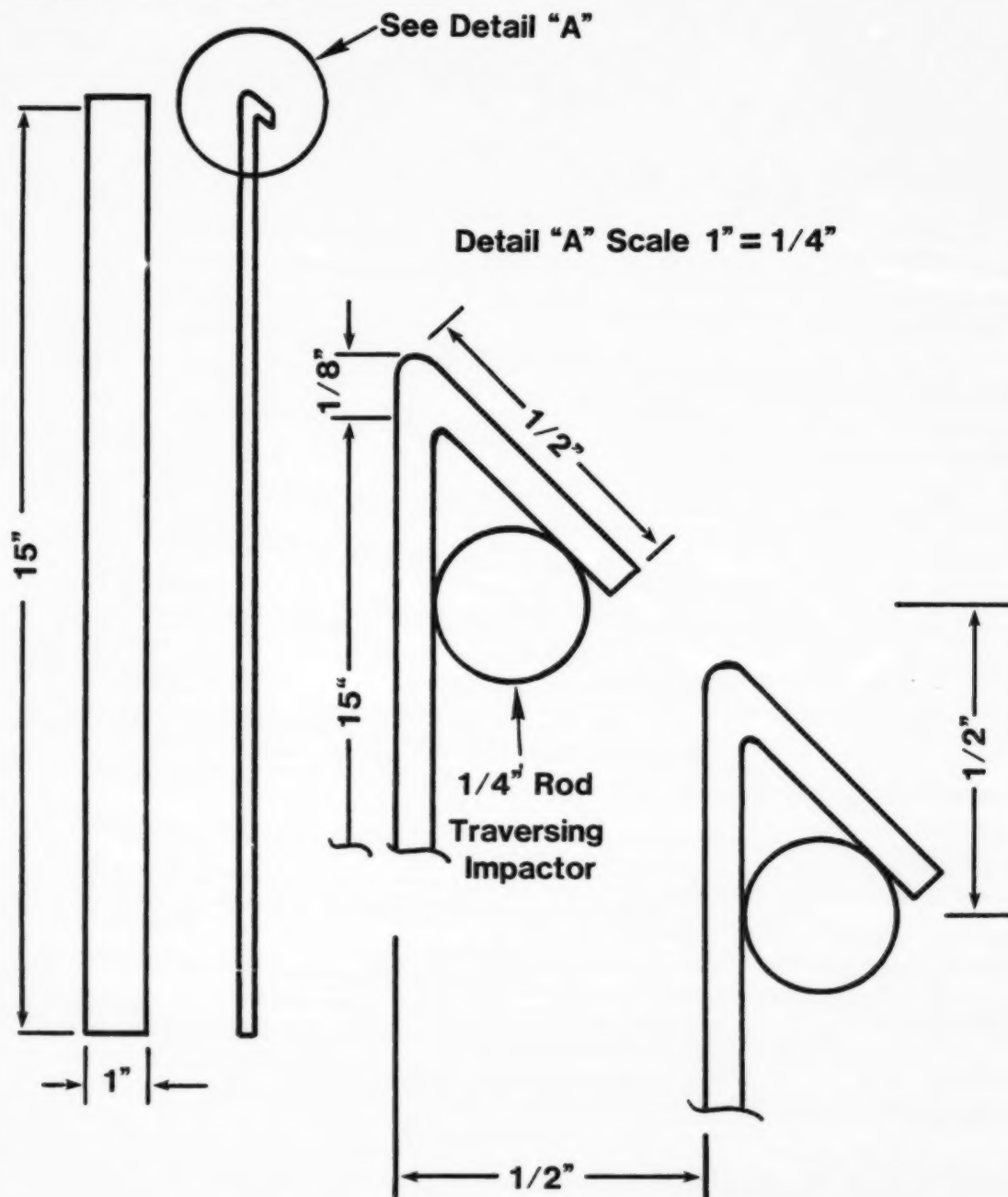


Figure A-4 Impactor Blades

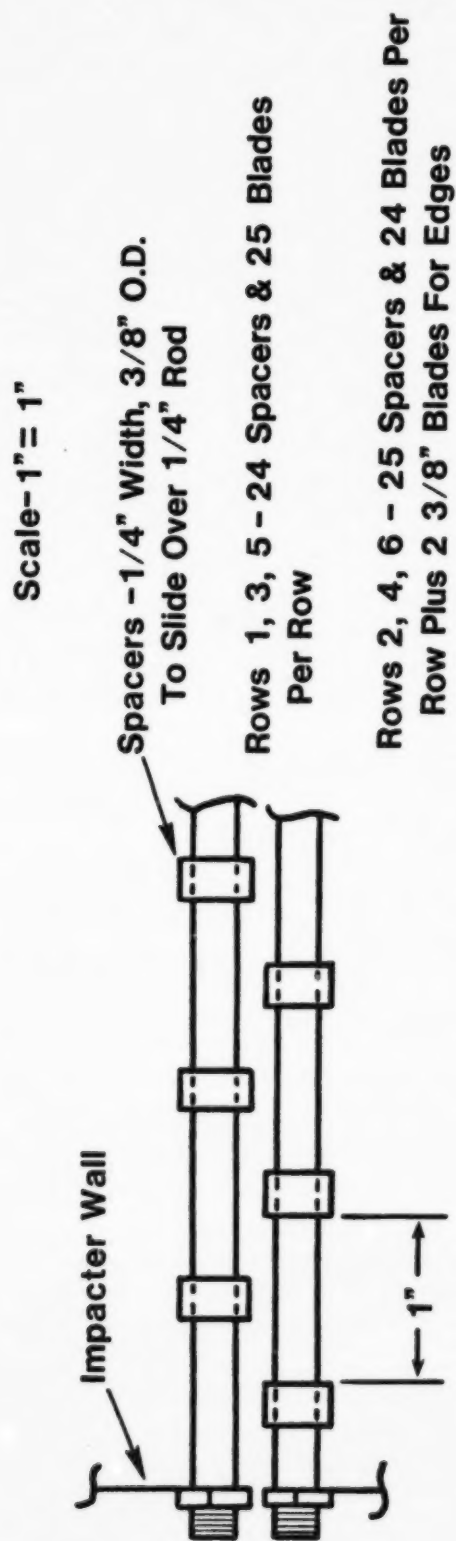


Figure A-5 Traverse Rod & Spacer Details

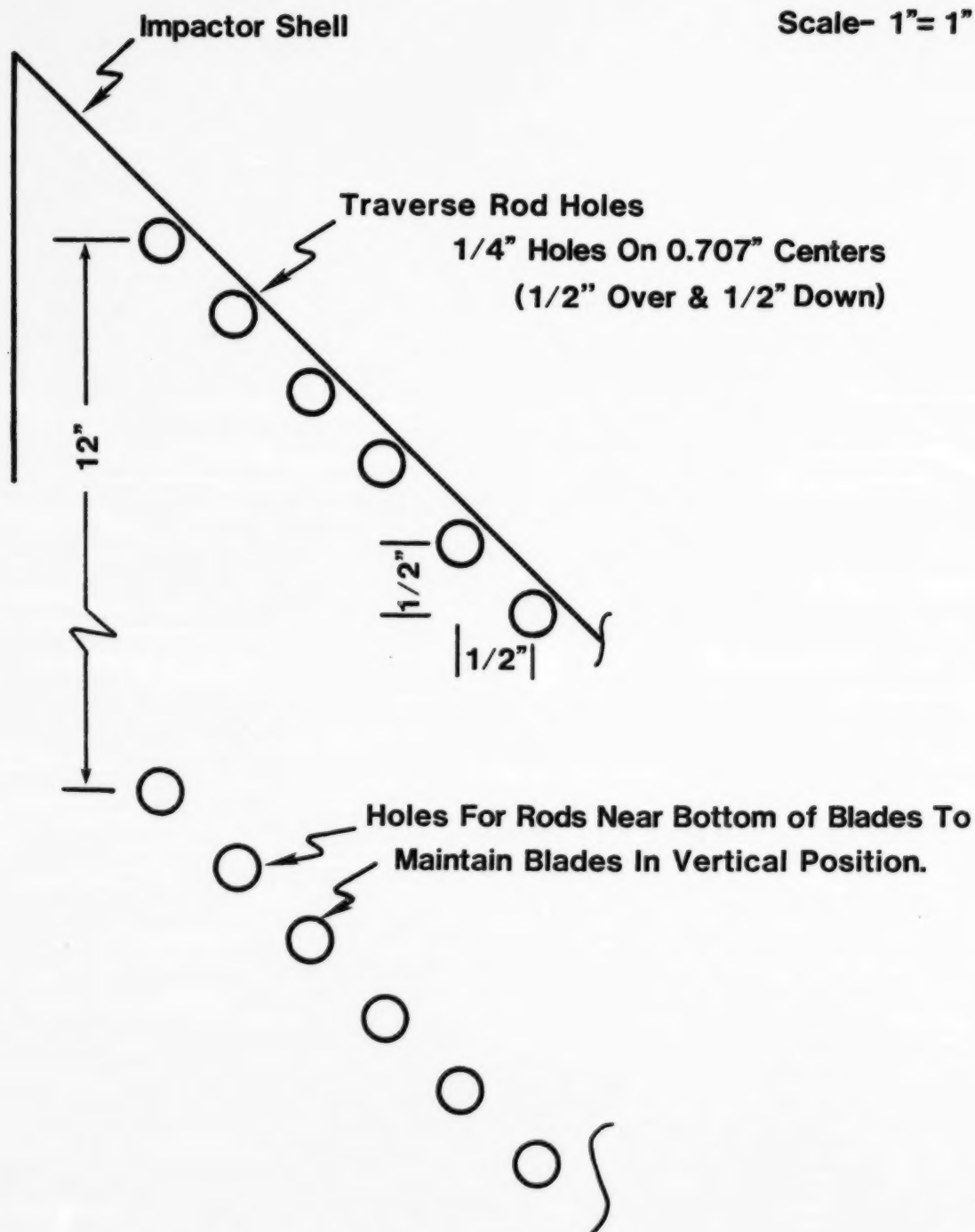


Figure A-6 Traverse Rod Holes

APPENDIX B
SI-U.S. CUSTOMARY UNITS CONVERSION FACTORS

To convert from:	To:	Multiply by:
pounds	kg	0.454
inches	m	2.54×10^{-2}
micron	m	1.00×10^{-6}
gallons	m ³	3.79×10^{-3}
lb/ft ³	kg/m ³	16.0
cfm	m ³ /s	4.72×10^{-4}
ft/min	m/s	5.08×10^{-3}
inches of water pressure	Pa	2.49×10^2
psi	Pa	6.89×10^3
watt	J/s	1.
ton (refrigeration)	J/s	3.52×10^3
°F	K	$(t^{\circ}\text{F} + 460)/1.8$